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MOISTURE PROTECTIVE COATINGS FOR ALKALI HALIDE IR OPTICS

F. Yamagishi and L. Miller

Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, CA 90265

July 1980

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1 November 1979 through 30 April 1980
ARPA Order No. 3833

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The objective of this program is to develop plasma-polymerized films that are transparent, moisture-protective coatings to increase the lifetime and durability of press-forged KBr IR optical elements.			
Reaction conditions have been established that yield uniform films of plasma-polymerized ethane (PPE) on polished and etched single-crystal KBr flats. On polishing, polycrystalline KBr formed			

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localized grains that could not be removed by chemical etching with HBr. Surface passivation could be achieved with a plasma of CF₃Br or CF₂Cl₂. An air leak in the reactor caused extensive corrosion problems with CF₃Br. Repairing the leak eliminated the corrosion problem. On a concurrent IR&D program, we are developing a new surface passivation method for NaCl using a non-IR-absorbing surfactant. PPE deposited on this treated surface afforded protection from damage by 98% relative humidity for 11 days. It is important to eliminate trapped free radicals in the film to reduce the chance of forming a more polar material caused by reaction of the radicals with atmospheric oxygen. Treating the film briefly with a hydrogen plasma quenched the radicals, but the method was no more effective than the present method of storing under an ethylene atmosphere. The use of DEHA, a two hydrogen atom donor, is being studied in an IR&D program as a radical quencher and may be applied to this program if it proves successful. HRL has purchased a relative humidity chamber to replace the saturated salt solutions used presently. This will allow for better control of humidity and ambient temperature. A holder for KBr flats and press-forged KBr is being designed, and prototypes, which use a water-tight sealant, are being tested. Environmental and durability tests according to MIL-STD-810B can be carried out at Hughes Aircraft Company, with the exception of fungus testing. NVEOL has agreed to changes in the test procedure for Salt Sea Atmosphere and Severe Humidity.

The statement of work will need to be modified if a variety of halo-carbon reactants for in situ surface passivation, thin film surfactants for surface passivation, DEHA for free radical quenching, and specific environmental testing according to MIL-STD-810B are to be included in this contract effort. These changes will not affect the cost of the contract.

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FOREWORD

This contract is administered by the Defense Advanced Research Projects Agency, Materials Science Office. Maj. Harry V. Winsor is the DARPA project engineer. Mr. Robert Dockery is the contract monitor for the Night Vision and Electro-Optics Laboratories, Ft. Belvoir, VA.

The program manager is Dr. Frederick G. Yamagishi, head of the Plasma Chemistry Group of the Device Materials Section headed by Dr. Leroy J. Miller. Professional assistance was provided by Ms. Diana D. Granger. Technical assistance was provided by Mr. Thomas S. Taylor and Mr. Willis H. Smith, Jr.

Work was performed in the Exploratory Studies Department of Hughes Research Laboratories under the management of Dr. Donald H. Close.

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SECTION I

INTRODUCTION

For the past three and one-half years, Hughes Research Laboratories (HRL) has been carrying out an internally funded program on IR protective coatings. HRL has an extensive background in IR materials, anti-reflection coatings, polishing, surface analysis, and, more recently, in protective coatings for alkali halide windows. Much of this expertise was used during this reporting period.

Alkali halides are attractive materials for IR detector and laser systems, particularly for use in the far-IR (8- to 12- μm) range, because of their transmissivity and relatively low cost. However, these materials are highly sensitive to moisture, which rapidly attacks the surface and degrades the optical quality. Furthermore, since the materials are soft and fragile, the optical components must be handled and cleaned carefully. Thus there is a need for protecting the surface of alkali halide optical elements from damage caused by exposure to uncontrolled environments and for increasing their durability so that they can be conveniently handled during installation and maintenance.

During our internally funded program on IR protective coatings, we investigated the use of polymeric materials prepared in a low-pressure, rf-generated glow discharge. This process is commonly called plasma polymerization. Polymers can be prepared conveniently in a plasma as thin, pinhole-free, adherent and conforming films. Their utility is enhanced by their hydrophobicity and their high crosslink density, which minimizes their permeability to water. A plasma polymer derived from ethane, which cannot be polymerized by any other method, is highly transparent in the 8- to 12- μm range. Thus, this material is a likely candidate for a low-absorption, moisture-protective coating.

Our investigation showed that the nature of the substrate surface is of paramount importance to enable plasma-polymerized films to act as effective protective coatings. For NaCl, extended lifetimes under high-humidity conditions required preparing a smooth, passivated surface by chemically etching with hydrochloric acid before depositing

the polymer film. On an internally funded program, we have developed an effective *in situ* surface-passivation method that uses the active species formed in a plasma derived from chlorofluorocarbon gases. We believe that the chlorine and/or fluorine atoms replace hydroxide groups on the crystal surface, and that these OH groups, if not replaced, are responsible for binding to the surface and accelerating its attack. Since we carry out both the passivation and the coating deposition in the same reactor, the passivated surface is not exposed to ambient conditions before the film is deposited. A plasma-polymerized ethane (PPE) protective coating deposited on a passivated NaCl window provided protection from damage by water vapor at 98% relative humidity (RH) for at least 100 hr, at which point the testing was arbitrarily stopped. The demonstration of tremendously increased lifetimes provided by the approaches taken in our IR&D research shows the promise of plasma-polymerized films as protective coatings for alkali halide lenses and windows.

Under a DARPA contract, Honeywell recently developed low-cost, press-forged KBr optical elements. These optics are being considered as a replacement for the internal ZnSe color-corrector element in FLIR systems being developed by the Night Vision and Electro-Optics Laboratories. The objective of our program is to develop plasma-polymerized films that are transparent and moisture protective and to use these to increase the lifetime and durability of press-forged KBr IR optical elements. It is anticipated that these optical elements will be enclosed in military FLIR systems under partially controlled atmospheric conditions but will be exposed occasionally to uncontrolled conditions during maintenance operations. Plasma-polymerized films will protect these elements from damage when the FLIR system is opened to humid environments.

SECTION 2

SUMMARY

This report details the progress made on a contract entitled "Moisture Protective Coatings for Alkali Halide Infrared Optics" under DARPA sponsorship with technical monitorship by the Night Vision and Electro-Optics Laboratories. It covers the first six months investigation of a 14-month program. The objective is to develop plasma-polymerized films that are transparent and moisture-protective and to use these to increase the lifetime and durability of press-forged KBr IR optical elements. This section summarizes progress by task. More detailed methods and results are presented in Section 3, which includes plans for future work on each task.

A. TASK 1: SURFACE PREPARATION AND REACTION CONDITIONS

Reaction conditions have been optimized to yield uniform films of PPE on polished and passivated single-crystal KBr flats. We found that commercially available polycrystalline KBr flats could not be polished without localized grains forming that could not be removed by chemical etching with HBr.

B. TASK 2: SURFACE PASSIVATION

Initial experiments with CF_3Br resulted in electrode corrosion and poor film quality. Subsequently, a small leak was discovered in the reactor system. Its repair eliminated the corrosion problem. We are continuing to optimize the method for CF_3Br as well as for CF_2Cl_2 . In a related IR&D program, a surface passivation method using a non-IR-absorbing surfactant is being developed. PPE deposited on a treated NaCl surface protected the window from damage by 98% RH for 11 days. We are suggesting that this method should be applied under this contract and that there should be an appropriate modification to the statement of work.

C. TASK 3: EXPANSIVE STRESS RELIEF

Expansive stress is commonly manifested by cracks in the film. Thus far, cracks have not been a problem with PPE on KBr.

D. TASK 4: FREE RADICAL QUENCHING

Free radical quenching was accomplished with hydrogen atoms derived from a hydrogen plasma, although this was no more effective than storing the freshly deposited film under ethylene, which is the method used currently. We are investigating in an IR&D program the use of N,N-diethylhydroxylamine, which is a two-hydrogen-atom donor, as a radical quencher. Should this be effective, the statement of work would need to be modified.

E. TASK 5: THICKNESS EFFECTS

Thickness and transmission are evaluated with each sample. We currently use a thickness of $\sim 5 \mu\text{m}$, which results in a 3 to 5% transmission loss (from the uncoated KBr flat) in the 8- to $12-\mu\text{m}$ region.

F. TASK 6: IR ABSORPTION MINIMIZATION

There has been no direct progress on this task aside from routine spectral measurements. Progress in this area is related to progress in Task 4, since the problem here is related directly to the concentration of residual free radicals.

G. TASK 7: MOISTURE RESISTANCE OF COATED SAMPLES

HRL recently purchased a relative humidity chamber to replace the saturated salt solutions used presently for 88% and 98% RH. Both RH and temperature can be controlled, and all further moisture testing will be done in this chamber. We are designing a holder, which will use a water-tight sealant, for KBr flats and press-forged KBr.

H. TASK 8: ENVIRONMENTAL AND DURABILITY TESTS

We determined that the required environmental and durability testing can be done within Hughes Aircraft Company, with the exception of fungus testing. However, several local laboratories can do fungus testing according to MIL-STD-810B. NVEOL has agreed to the following changes in the test procedure (MIL-STD-810B):

- Salt Sea Atmosphere. Method 509, Proc. I for 48 hr instead of 120 hr
- Severe Humidity. Method 507, Proc. I instead of Proc. II.

To implement the specific requirements for this testing will require modifying the statement of work. The three necessary changes (mentioned above) will have no effect on the cost of the contract.

SECTION 3

TECHNICAL DISCUSSION

This section discusses, task by task, the problems and solutions encountered during the first six months of the contract. Work yet to be completed is also discussed.

A. SURFACE PREPARATION AND REACTION CONDITIONS

Task 1. Establish reaction conditions to prepare plasma-polymerized ethane on polished and passivated KBr windows and press-forged KBr optics. Ascertain polymer structure by quantitative carbon-13 nuclear magnetic resonance spectroscopy.

In prior work, we had used sodium chloride substrates to develop our technique for using plasma-polymerized films as moisture-protective coatings. Unfortunately, this technology on the surface preparation of polycrystalline NaCl was not entirely applicable to polycrystalline KBr. We found that commercial KBr (Harshaw) could not be polished without large-grain domains forming. After grinding by standard methods, various methods of polishing were used on a total of 17 samples: (1) polishing on Polytex Supreme with Linde A in isopropyl alcohol; (2) polishing on pitch with Linde A in ethylene glycol; and (3) polishing with black or red rouge on a variety of backing cloths. In no case were the domains eliminated. Furthermore, chemical etching with 48% HBr, which is a method described in the literature for repairing grinding damage at the surface, proceeded at different rates in these domains and the remaining area. The etching did not completely remove the grains ($\sim 0.5 \mu\text{m}$ in diameter), and the optical flatness was degraded severely. A film of PPE on these surfaces, which were passivated with plasma-generated halogen atoms, afforded protection at 88% RH for no more than 3 hr.

We have eliminated the problem by using single-crystal KBr (Harsaw, crystallographic face unknown). No grain boundaries were found on grinding, and chemical etching with 48% HBr for ~ 30 sec yielded a smooth, wet-looking surface.

Reaction conditions for the deposition of uniform films of PPE on these smooth surfaces have been established, although the deposition rate is fairly low ($\sim 0.25 \mu\text{m}/\text{hr}$). Twenty six experiments were carried out, in which pressure and flow rate were varied while rf power was held constant. The deposition rate was calculated by measuring the thickness of the film (Dektak FLM surface profile measuring system). Although higher deposition rates would obviously be desirable, we found that an oil film was obtained at rates above $\sim 0.3 \mu\text{m}/\text{hr}$. Those PPE films that were of high quality visually were studied spectrophotometrically. The reaction conditions that produced the highest quality films were chosen for this work. Periodically we make calibration runs on glass slides to verify the reproducibility of film quality under a given set of conditions.

We plan to obtain carbon-13 NMR spectra on PPE at Colorado State University's Regional NMR Center (an NSF-sponsored state-of-the-art instrument facility). Dr. G.E. Maciel, the director of the center, is eager to carry out these experiments, but their magic angle-cross polarization instrumentation (needed for fine-line solid-state spectra) is still in the construction stage.

B. SURFACE PASSIVATION

Task 2. Optimize our *in situ* surface-passivation method for KBr using bromine-containing halocarbon reactants, and, using Auger spectroscopy, study its effectiveness.

We had found previously that halogen atoms generated from inert Freon gases effectively passivated the surface of NaCl. We believe that halogen atoms replace surface hydroxide groups. One advantage of this method is that it is done under vacuum in the reactor just before the plasma-polymerized film is deposited, thus voiding contamination problems. On this program, we initially used CF_3Br for the surface passivation of KBr. However, we found that this material caused electrode corrosion, presumably because Br_2 formed in the plasma. The PPE films deposited thereafter were inferior, as evidenced by their cloudy

appearance and large refractive index change, caused possibly by incorporation of halogen, decomposition fragments of CF_3Br , or corrosion products. The corrosion problem was traced to a small air leak in the reactor system. After the leak was repaired, we found that CF_3Br could indeed be a useful starting material for passivation. Although we did not investigate the specific role of air, we speculate that excited oxygen and nitrogen molecules or atoms interact with the excited species in the plasma. Bromine may be an ultimate product of this interaction, which would account for the electrode corrosion. Also, excited nitrogen and/or oxygen could interact with CF_3 radicals to form a species that may be polymerizable in a plasma. A polar film of this type would be expected to have a significantly different index than PPE. Therefore, the deposition of PPE would actually yield a composite having an index different from that expected. We are continuing to optimize conditions for maximum passivation with CF_3Br . During the corrosion problem, we investigated the utility of CF_2Cl_2 for surface passivation. This material did not cause corrosion and did not affect the optical properties of the KBr. We are continuing to optimize reaction conditions for this compound as well as for CF_3Br .

On a concurrent internally funded program, we are developing another surface-passivation technique, one not covered by the statement of work for this program. This technique uses a surfactant which is applied as a very thin film and is therefore transparent in the IR. Treating the surface of NaCl with this surfactant and then depositing PPE afforded protection from damage by 98% RH for 11 days. During this period, no cracks or bubbles in the film were observed. Unfortunately, no spectral data were obtained on this sample because the film was torn when it was removed from its holder. Methods to eliminate this holder are discussed below in Section 3.G (Task 7).

We use an O-ring cylinder, piston-type holder (see Figure 1) for RH testing so that only one side of the alkali halide window need be coated. The coated window is placed at the bottom of the cylinder, and the film is exposed to high RH through a hole in the piston. A

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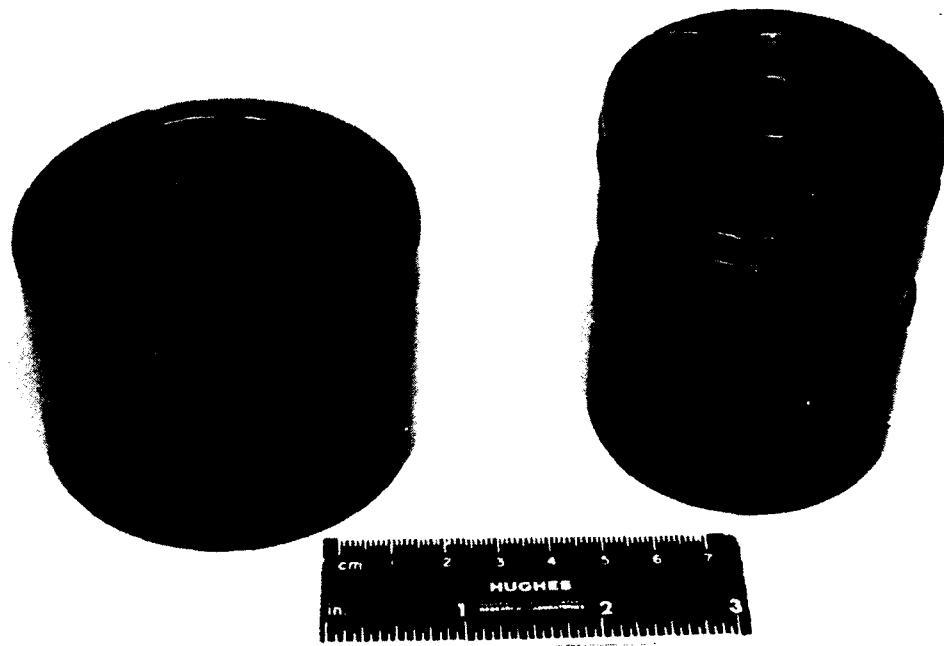


Figure 1. O-ring holder for humidity testing.

A seal at the hole is provided by a Buna rubber O-ring, which is compressed against the coated alkali halide window. When the holder is disassembled, the film often is torn by this O-ring, either because of sticking or because of a twisting at the interface. Another explanation could be that percolation of water through the film reduces adhesion at the film-surface interface, which would allow the film to be torn.

To include work on the application of this surfactant, we will notify DSSW requesting a change in the wording of this task in our original statement of work to read:

"Optimize our *in situ* surface-passivation method for KBr using a variety of halocarbon reactants, and, using Auger spectroscopy, study their effectiveness; and investigate other novel passivation techniques, which may not be *in situ* methods, such as thin layer surfactants."

This change would allow implementing the surfactant method for use in this contract effort. As mentioned above, we plan to continue to optimize reaction conditions for our *in situ* surface-passivation method.

The Auger spectrometer has been dedicated to another, nearly completed project. When it becomes available, we will use it to monitor the level of effectiveness of our *in situ* methods.

C. EXPANSIVE STRESS

Task 3. Study the relief of expansive stress in the films by annealing and by decreasing the deposition rate using pulse-mode operation.

In the past, film failure from exposure to high humidity caused by expansive stress was manifested by large cracks in the film. This has not been seen in films on KBr, which implies that expansive stress is not a problem on KBr. Therefore, we have not studied annealing or pulse-mode deposition. Should film failure due to expansive stress eventually be observed, we will implement these methods.

D. QUENCHING FREE RADICALS

Task 4. Investigate the quenching of trapped free radicals with hydrogen atoms derived from a hydrogen plasma; study the decrease of free-radical concentration by pulse-mode deposition and post-heating; and determine the effectiveness of these techniques by electron spin resonance spectroscopy.

It is known that long-lived free radicals are trapped in plasma-polymerized films. For the film to retain maximum hydrophobicity, the free radicals must be efficiently quenched so that no polar oxygenated sites can form on the surface or in the bulk by exposure to the atmosphere. We presently allow the residual free radicals in PPE to act as initiating agents for the chain-reaction polymerization of ethylene. Although this method works reasonably well, it is, as expected, neither efficient nor completely effective, as shown by the data in Table 1. Treating PPE with ethylene and then exposing the film to dry air resulted in the reaction of oxygen with remaining trapped radicals after 56 days, as shown by the formation of a carbonyl group which was observed in the IR spectrum. A chain reaction polymerization generates a new free radical in each step as the chain grows, and eventually the polymeric radical must be quenched by reaction with another radical or a chain transfer agent. With plasma-polymerized films, termination can only occur by the radicals of two growing chains coming into contact with each other. This is not a very efficient process since the polymeric radicals are trapped within a solid matrix, where they are often isolated. Furthermore, ethylene can permeate the polymer matrix and partially react with radicals below the surface, causing disclinations in the polymer structure, localized swelling, and increased expansive stress.

We treated PPE with hydrogen atoms derived from a hydrogen plasma, but only for 5 min. Comparison of the effectiveness of this method with that of storing the film under an ethylene atmosphere is shown in Table 1. Three KBr flats were coated with PPE and treated as shown. Each was removed from the reactor and stored in dry air. They were monitored periodically for the formation of a carbonyl group, which

Table 1. A Comparison of Free Radical Quenching Methods

Treatment	Time for C=O Appearance, Days
Stored under O ₂	37
Stored under ethylene	56
H ₂ plasma/stored under H ₂	52

shows a strong absorption at $\sim 5.8 \mu\text{m}$ in the IR. We found that treating PPE with a hydrogen plasma for 5 min did quench free radicals, but the method was no more effective than storing under ethylene. Storing under a hydrogen atmosphere was not effective.

We have investigated on an IR&D program the use of N,N-diethyl-hydroxylamine (DEHA), a two-hydrogen-atom source, as a free radical quencher. Figure 2 shows the chemical reaction; in this case, R is a fragment of the PPE film. An efficient technique for exposing the film to DEHA has not been optimized, but we feel that this could be a useful method.

We plan to investigate further the use of hydrogen plasmas for extended periods of time and to refine experimental conditions for DEHA. Use of DEHA, however, would require a change in the statement of work. We will request that DSSW change this task to read:

"Investigate the quenching of trapped free radicals with hydrogen atoms derived from a hydrogen plasma as well as from other hydrogen atom sources not necessarily derived from a plasma; study the decrease in free radical concentration by pulse mode deposition and post-heating; and determine the effectiveness of these techniques by electron spin resonance spectroscopy."

The combination of continuous mode deposition with pulse-mode deposition for the outermost layer of the film will be investigated. We anticipate that the pulse-mode-prepared polymer will have a lower concentration of free radicals than the polymer prepared continuous mode. We feel that the lower number of radicals can be more completely

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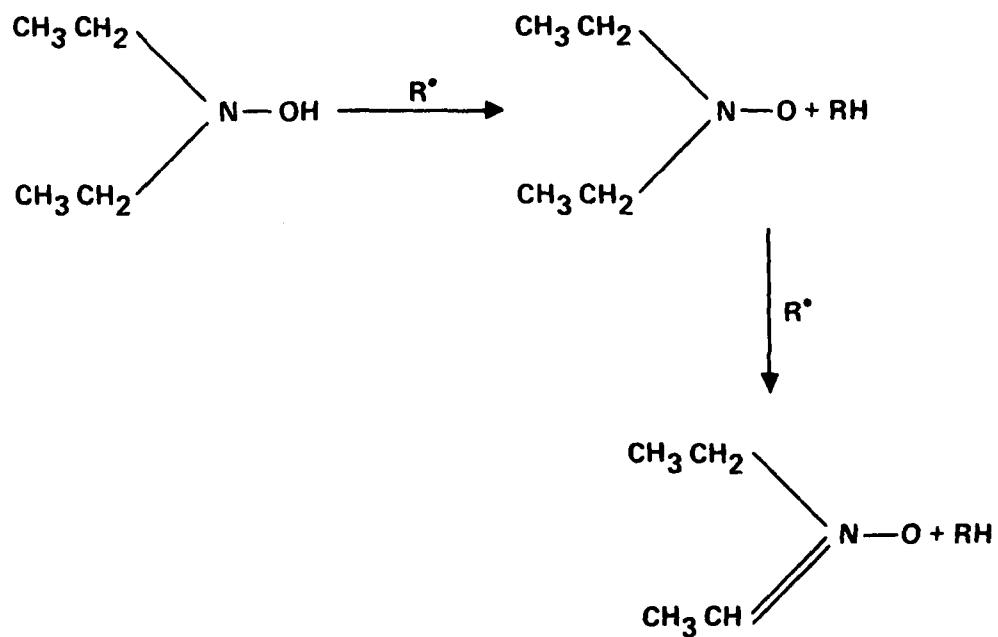


Figure 2. Free radical quenching scheme for *N,N*-diethylhydroxylamine.

quenched and that the radical-free outer layer will "bury" the radicals trapped in the underlying layer. We have made arrangements with Dr. K. Fang of the Department of Chemistry at UCLA to obtain electron spin resonance spectra on a service basis to monitor more quantitatively the effectiveness of the various quenching methods.

E. THICKNESS EFFECTS

Task 5. Determine the optimum thickness for adequately coating surface irregularities while having minimum interference with the optical performance of the window. Establish conditions for uniform deposition on both surfaces and edges of a plano-concave structure.

The effect of thickness on moisture protection and transmission is evaluated with each sample. Presently, we use a thickness of $\sim 5 \mu\text{m}$, which corresponds to $\sim 3/4$ of a wavelength at $10.6 \mu\text{m}$ and which, therefore, should provide a maximum in transmission at that wavelength. We plan to continue thickness evaluations. Work related to this task on press-forged KBr will begin as soon as samples become available.

F. IR ABSORPTION MINIMIZATION

Task 6. Evaluate methods for minimizing absorption in the 8- to $12-\mu\text{m}$ range by employing reaction conditions that minimize carbon-carbon double bond formation and by removing residual free radicals to prevent the formation of carbon-oxygen functions in the film. Monitor the absorption by IR and ATR spectroscopy and by laser calorimetry at $10.6 \mu\text{m}$.

We have made no direct progress on this task other than obtaining routine IR spectra on each sample. Progress in this task can be correlated with progress in Task 4 since the problem here is directly related to the concentration of residual free radicals.

C. MOISTURE RESISTANCE OF COATED SAMPLES

Task 7. Measure moisture resistance of plasma-polymerized films on KBr by exposure to relative humidities ranging from 84 to 100% (specifically 84, 88, 92, 98, 100% RH), with concurrent temperature cycling between -54° and 95°C. Our goal is a lifetime of at least 200 hr in 88% relative humidity at 20°C.

An RH chamber has been purchased with company funds. We previously had used saturated salt solutions for 98% RH (cupric sulfate pentahydrate) and 88% RH (potassium chromate). Both solutions are rated at 20°C. The temperature in the laboratory is usually higher than that and is rarely constant. Our new chamber, which is currently being evaluated, will control the temperature to $\pm 0.5^\circ\text{C}$ and the RH to $\pm 2\%$. All routine testing will now be done in this chamber.

We are currently testing a variety of water-tight sealants to replace the O-ring holder described in Section 3.B (Task 2). The sealant must not only be impervious to moisture penetration for extended periods but must be easily removed without the optical quality of the surface being affected. Seven experiments using collodion, paraffin, clear fingernail polish, and two types of stop-off lacquers (used in electropolishing) all met with only marginal success. The most promising material is a plastic coating called PDS (Plasti-dip International). After six experiments, this material met the requirements mentioned above. We plan to investigate RTV-60 (General Electric), which is used to mount the color corrector lens into the holder of the imager, for its sealing capability and compatibility with PPE on KBr.

H. ENVIRONMENTAL AND DURABILITY TESTS

Task 8. Evaluate the adherence and abrasion resistance of the plasma-polymerized ethane films on press-forged KBr as specified in the appropriate quality assurance provision sections of MIL-Specifications MIL-C-675, MIL-C-675A, MIL-M-13508C, and MIL-STD-810B.

The environmental and physical durability tests on the coated optical element according to the pertinent methods and procedures of MIL-STD-810B as listed in the development specification for the optical imager (Spec. No. B2-28A050104A) can be carried out within Hughes Aircraft Company, with the exception of fungus testing. Furthermore, NVEOL has agreed to the following changes in the test procedure (MIL-STD-810B):

- Salt Sea Atmosphere. To test by Method 509, Procedure I for 48 hr rather than 120 hr
- Severe Humidity. To test by Method 507, Procedure I instead of Procedure II.

There are several local laboratories that have assured us that they can perform fungus testing according to MIL-STD-810B.

To implement the environmental testing outlined in Specification B2 will require modifying the statement of work. We will request DSSW to change this task to read:

"Test and evaluate the adherence of plasma polymerized ethane films on press-forged KBr for compliance with the environmental and durability specification requirements according to the Development Specification for Alkali Halide Lens Elements with Moisture Protective Coatings. Specifically:

- A. Moderate abrasion — according to MIL-C-675B
- B. Adhesion — according to MIL-C-675B
- C. Humidity — according to MIL-C-675B
- D. Salt sea atmosphere — according to MIL-STD-810B, Method 509, Procedure I for 48 hr
- E. Severe humidity — according to MIL-STD-810B, Method 507, Procedure I (instead of Procedure II)
- F. Fungus — according to MIL-STD-810B, Method 508, Procedure I."

The three statement of work changes outlined in this report will have no effect on the cost of the contract.

We expect to start environmental testing in ~3 months.

I. ASSESSMENT OF PROGRESS

Significant progress has been made to date on achieving the objectives of this contract. We are in a position to prepare uniform plasma-polymerized films on smooth KBr flats, and we are making excellent strides in the areas of surface passivation and free radical quenching. We will be fine-tuning our efforts towards addressing the more detailed aspects of our proposal such as determining optimum thickness for maximum transmission and protection, investigating structure by various spectroscopic studies, and possibly relieving expansive stress.